2

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MICROSTRUCTURAL STABILITY AND CREEP RESPONSE OF A NEW Al-Fe-W-Si ALLOY

Dr. John Benci and Dr. William Frazier
Air Vehicle and Crew Systems Technology Department (Code 60634)
NAVAL AIR DEVELOPMENT CENTER
Warminster, PA 18974-5000

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The microstructural stability and creep properties of a new aluminum alloy for potential aerospace applications at service temperatures up to 700°F (371°C) were evaluated. The Al alloy composition is 11.50% Fe, 3.75% W and 2.25% Si by weight. The material was rapidly solidified via planar flow casting, vacuum hot-pressed, and then extruded into bar form. The room temperature yield strength and ultimate tensile strength are 603 MPa and 626 MPa, respectively, with an 11.3% elongation and a 33.0% reduction of area at fracture. A series of creep experiments were performed on this alloy at 700°F (371°C), and the results were compared to alloys FVS0812 (8009) and FVS1212. A change in creep deformation mechanism was discovered at a stress level of approximately 85 MPa defining the maximum allowable stress for components made from this material. The microstructural stability was evaluated by aging samples at either 700°F (371°C) or 900°F (482°C) and measuring the average grain and dispersoid sizes as a function of aging time with transmission electron microscopy. Upon aging at 371°C, the room temperature hardness did not change even after times greater than 1000 hrs., indicating the microstructure is stable at this temperature. The hardness decreased slightly during 482°C aging over a period of 670 hrs. indicating minor microstructural changes (e.g., dispersoid coarsening) were occurring over long periods of time.

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CONTENTS

FIGURES

TABLES

Acknowledgements

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Introduction

High temperature aluminum (HTA) alloys have been the subject of intense research and development activity in the aerospace community. The objective of this work is to enhance the performance and reduce the cost of advanced aircraft. This would be accomplished by replacing components made from heavy and difficult to machine titanium and steel alloys, used at temperatures between 150 and 375°C, with HTA alloy components.

The Navy, under the Intergrated High Performance Turbine Engine Technology (IHPTET) Program, is currently pursuing the development of HTA alloys for applications to 700°F (371°C). One approach has been to augment the properties of the "FVS1212 type" alloys, produced by Allied Signal, through compositional changes, e.g., replacing V with W.

The excellent ambient and elevated temperature strength of FVS1212 is attributable to the alloy's fine grain size (0.5 microns), and to the high volume fraction (36 to 37 vol. %) and fine size (30-80nm) of the metastable silicide dispersoids, viz., alpha-$Al_{12}(Fe,V)_3Si$ [1,2]. The silicide is isostructural with $Al_{15}Mn_3Si$; it is bcc (Im3) with 138 atoms per unit cell [3]. The addition of V stabilizes the alpha-$Al_{12}(Fe,V)_3Si$ structure sufficiently so that during planar flow casting it forms in preference to the thermodynamically more stable hexagonal Al_8Fe_2Si and monoclinic Al_3Fe phases [4]. W and other transition elements are able to substitute for V in the silicide's structure; the composition of the silicide phase is known to affect its coarsening rate [5]. W was added in order to curtail the coarsening rate and enhance the modulus of the silicide. The low diffusivity, limited solid state solubility, and high melting temperature of W made it attractive for this application.

The focus of this investigation was two-fold: (i) assess the creep behavior of a W bearing alloy at 371°C (700°F), and (ii) assess the effect of stress on microstructural coarsening. The combined effect of stress and temperature is known to alter the coarsening behavior of HTA alloys. For example, creep significantly increased particle coarsening in Al-Fe-Ce alloys by activating dislocation and grain boundary diffusion mechanisms [6]. At 550°C, alloy FVS1212 also experiences rapid growth of the alpha-$Al_{12}(Fe,V)_3Si$ dispersoids located at grain boundaries [1].

Experimental

One of the experimental, advanced, aluminum alloys developed under the IHPTET program by Allied Signal was selected as the material to be used in this study. This alloy was designated 102. Alloy 102 was chosen because it had the best combination of room temperature properties, i.e., strength and ductility. This alloy contains approximately 11.50% Fe, 3.75% W, and 2.25% Si by weight. Alloy 102 has the following room temperature properties: Yield Strength - 603 MPa (87.5 ksi); Ultimate Tensile Strength - 626 MPa (90.8 ksi); elongation to failure - 11.3%; reduction of area at fracture - 33%. The density of this alloy is calculated to be approximately 3.1 gcm^{-3}.

Standard creep specimens were machined from alloy 102 and from two of Allied Signal's commercial alloys, FVS0812 (8009) and FVS1212, which have compositions, processing conditions, and microstructures similar to alloy 102. One sample each of FVS0812, FVS1212 and alloy 102 was creep tested at 371^{o}C (700^{o}F) with step increases in load over a large stress range. The strain in the samples was recorded as a function of time using a dual LVDT extensometer. The Young's moduli at room temperature and 371^{o}C were determined for these three alloys by increasing the load on the specimen in an abrupt manner and measuring the instantaneous elongation using lvdt's. Based on the results from the alloy 102 variable load creep test, a test schedule for further creep experiments on this alloy at 371^{o}C was formulated. The minimum creep strain rate of each sample was measured. Interrupting the tests at predetermined times allowed the evaluation of the time and stress magnitude effects on any stress induced microstructural changes.

Samples of alloys FVS0812, FVS1212 and 102, 10mm x 13mm x 3mm, were cut from extruded bar and used in the aging study. Samples of each alloy were placed in a furnace at 371^{o}C or 482^{o}C and aged for various times. The room temperature hardness of these samples was then measured as a function of aging time and temperature. TEM specimens were prepared from each sample for microstructural characterization. The dispersoid sizes and grain sizes were measured in the specimens examined.

Results

Creep Testing

One sample each of FVS0812, FVS1212 and alloy 102 was creep tested at 371^{o}C (700^{o}F) with step increases in load over a large stress range. The strain rate vs. applied stress data are tabulated in Table I. The creep response of the three samples at 371°C is shown graphically in fig. 1.

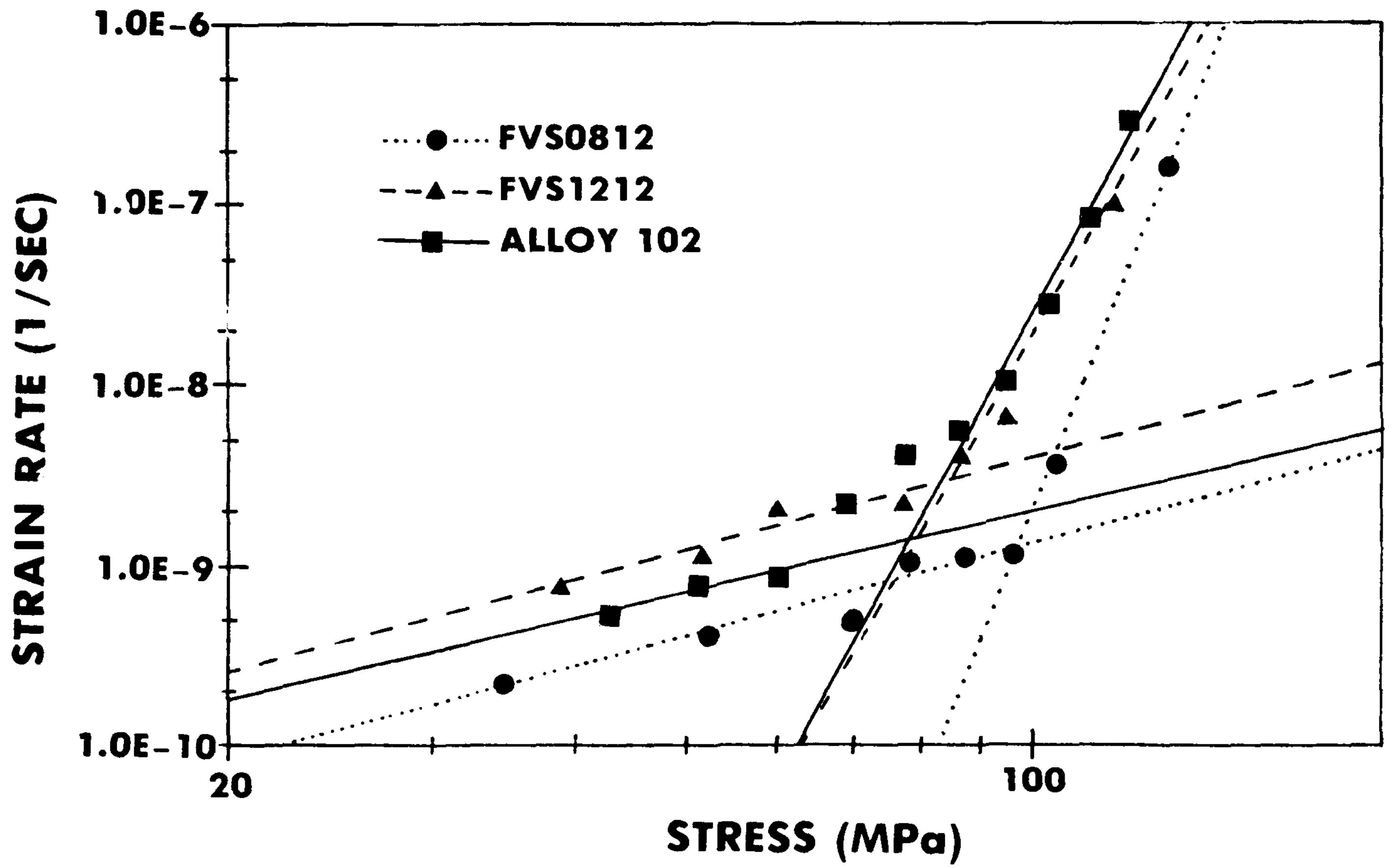


Figure 1 - Minimum creep rate as a function of applied stress.

The curves for all three samples exhibit a "knee" at about 80-100 MPa applied stress. The power law creep equation:

$$e_{min} = B\sigma^{n}$$

where e_{min} is the minimum creep strain rate, σ is the applied stress, and B and n are constants, was fit to the two segments of the curve for each sample. The values of n from the best fit curves for the three alloys are given in Table II. The "average" value of the stress exponent for the three alloys is about $n = 1.7$ for $\sigma < 80$ MPa and $n = 12$ for $\sigma > 90$ MPa. The creep behavior of alloy 102 at 371°C is fairly similar to that of alloy FVS1212.

Table I Minimum creep strain rate at 371°C as a function of applied stress from variable load creep tests.

FVS0812		FVS1212		102	
σ(MPa)	$e_{min}(s^{-1})$	σ(MPa)	$e_{min}(s^{-1})$	σ(MPa)	$e_{min}(s^{-1})$
34.79	2.33×10^{-10}	38.70	7.64×10^{-10}	43.00	5.31×10^{-10}
52.19	4.11×10^{-10}	51.60	1.15×10^{-9}	51.60	7.84×10^{-10}
-----	--------	60.20	2.17×10^{-9}	60.20	8.75×10^{-10}
69.59	5.25×10^{-10}	-----	--------	68.80	2.24×10^{-9}
78.29	1.05×10^{-9}	77.40	2.28×10^{-9}	77.40	4.28×10^{-9}
86.99	1.14×10^{-9}	86.00	4.26×10^{-9}	86.00	5.80×10^{-9}
95.69	1.22×10^{-9}	94.60	7.12×10^{-9}	94.60	1.14×10^{-8}
104.4	3.81×10^{-9}	103.2	3.35×10^{-8}	103.2	2.90×10^{-8}
-----	--------	116.1	1.11×10^{-7}	111.8	9.01×10^{-8}
130.5	1.69×10^{-7}	-----	--------	120.4	3.18×10^{-7}

Table II: Values for the constant n in the power law creep equation from variable load creep tests.

Alloy	Low Stress n	High Stress n
FVS0812	1.72	16.11
FVS1212	1.71	11.45
102	1.50	11.90

The Young's moduli at room temperature and 371°C were determined for these three alloys by increasing the load on the specimen in an abrupt manner and measuring the instantaneous elongation using lvdt's. The moduli for alloys FVS0812, FVS1212 and 102 at room temperature are 84.1 GPa, 89.0 GPa, and 91.0 GPa, respectively, and are 63.4 GPa, 70.3 GPa, and 68.3 GPa, respectively, at 371°C.

Based on the results from the alloy 102 variable load creep test, a test schedule for further creep experiments on this alloy at 371°C was formulated. The test schedule is shown in Table III. The minimum creep strain rate of each sample was measured. Interrupting the tests at the indicated times allowed the determination of the time and stress magnitude effects on any stress induced microstructural changes. The 100 hour test with an applied stress of 110 MPa was initiated, however the sample failed after a test time of only 32.5 hrs. The 95 MPa test scheduled for 100 hrs. was therefore interrupted after 32.5 hrs. to maintain a constant test time for the two stress levels. The minimum creep strain rates of the six tests performed are shown under the heading of Constant Load in Table IV. The minimum strain rate results from the alloy 102 sample which was creep tested under variable load conditions are also presented in Table IV. The results from the two types of tests match fairly well. TEM specimens were made from each creep sample to determine time and stress magnitude effects on any stress induced microstructural changes.

Table III: Test schedule for creep experiments on alloy 102 at 371°C.

Applied Stress (MPa)	Test Time (hrs)
40.0	500
60.0	500
60.0	200
95.0	200
95.0	100 (32.5)
110.0	*100 (32.5)

* Sample failed after only 32.5 hours.

Table IV: Minimum creep strain rate of alloy 102 at 371°C as a function of applied stress.

CONSTANT LOAD		VARIABLE LOAD	
(MPa)	e_{min} (s^{-1})	(MPa)	e_{min} (s^{-1})
40.00	1.9×10^{-10}	43.00	5.31×10^{-10}
60.00	8.15×10^{-10}	51.60	7.84×10^{-10}
60.00	8.80×10^{-10}	60.20	8.75×10^{-10}
95.00	1.29×10^{-8}	68.80	2.24×10^{-9}
110.0	9.91×10^{-8}	77.40	4.28×10^{-9}
		86.00	5.80×10^{-9}
		94.60	1.14×10^{-8}
		103.2	2.90×10^{-8}
		111.8	9.01×10^{-8}
		120.4	3.18×10^{-7}

Aging Studies

Aging studies were conducted on alloy 102 to determine the suitability of this alloy for extended use at elevated temperatures. Alloys FVS0812 and FVS1212 were included in this study for comparison purposes. Samples of alloys FVS0812, FVS1212 and 102, 10mm x 13mm x 3mm, were placed in a furnace at 371°C (700°F) and aged for various times between 4 and 1024 hours. The samples were removed from the furnace at predetermined times and were air cooled. Hardness measurements were taken from each of the aged samples and from two unaged "as-received" samples of each alloy. The hardness measurements are tabulated in Table V as a function of aging time. The hardness of all three alloys does not change as a function of aging time at 371°C up to at least 1024 hrs. This result indicates that the microstructure is stable at this temperature. TEM measurements were made to verify this thesis. The hardness of alloy 102 (R_B 92.0) is slightly greater than alloy FVS1212 (R_B 89.2) which is harder than alloy FVS0812 (R_B 77.4).

Table V: Hardness of alloys as a function of aging time at 371°C

Aging Time	Hardness (Rockwell B)		
HOURS	FVS0812	FVS1212	102
0	76.7	89.1	91.2
4	-	-	92.1
8	76.4	89.0	92.1
16	77.2	89.3	92.1
32	77.7	89.3	92.0
64	78.0	89.3	92.2
128	78.2	89.6	91.8
256	76.8	89.2	92.1
512	77.9	89.4	91.6
768	77.8	89.0	-
1024	77.9	89.4	91.3

Samples of alloys FVS0812, FVS1212 and 102, 10mm x 13mm x 3mm, were placed in a furnace at 482°C (900°F) and aged for various times. The room temperature hardness of these samples was then measured. Table VI lists the hardness of these alloys as a function of aging time. The hardness of all three alloys decreased upon aging at 482°C, compared to no change in hardness when aged at 371°C (700°F). However, for alloy 102, the changes in hardness resulting from the 482°C aging were relatively small, even after 671 hours, indicating that the microstructure of this alloy is relatively stable even at this elevated temperature.

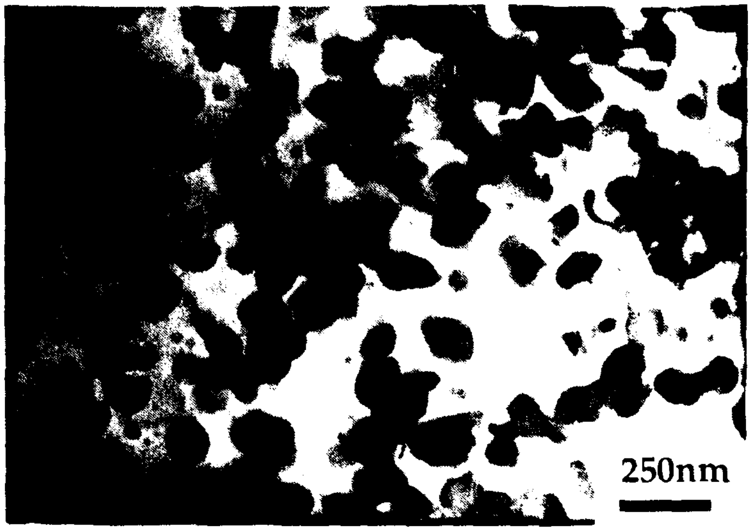
Table VI: Hardness of alloys as a function of aging time at 482°C.

Aging Time	Hardness (Rockwell B)		
HOURS	FVS0812	FVS1212	102
0	76.7	89.1	91.2
168	74.3	87.3	89.7
352	73.0	87.2	88.7
502	-	-	87.8
671	-	-	87.8

Transmission Electron Microscopy

Two micrographs showing the "average" or "typical" microstructure of alloy 102 are presented in fig. 2. Figure 2(a) is a micrograph of alloy 102 in the as-received

a)



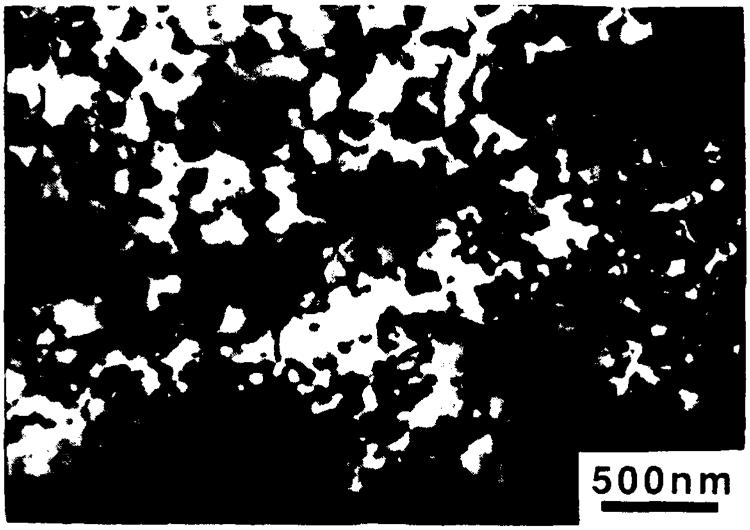
As-Received

b)

Aged for 128 hrs. at 371°C

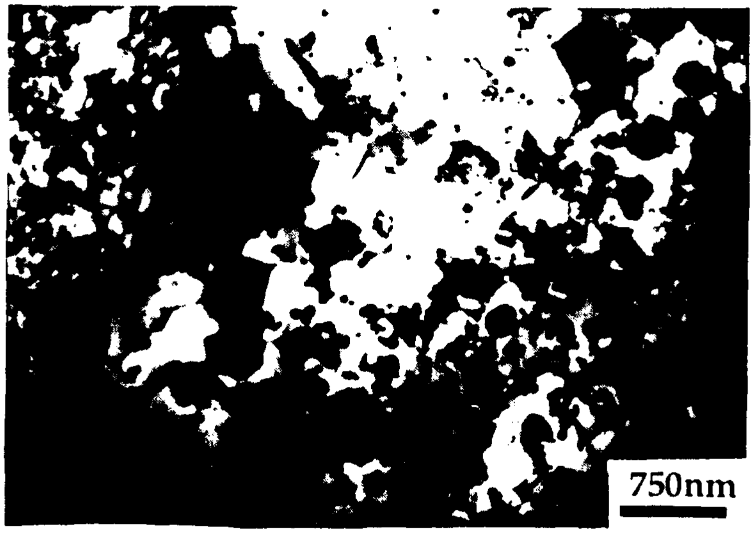
Figure 2 - TEM micrographs illustrating the "average" microstructure of alloy 102 in the (a) as-received condition, and (b) after aging for 128 hrs at 371°C.

a)



Bimodal Dispersoid Sizes

b)



Dispersoid Lean Region

Figure 3 - TEM micrographs illustrating the inhomogeneous microstructure of alloy 102 in samples aged for 128 hrs at 371°C. (a) Bimodal dispersoid size distribution and (b) dipersoid poor regions.

condition. The micrograph in fig. 2(b) is from a sample which had been aged at 371°C for 128 hrs. The average dispersoid size is about 0.08 microns in diameter and the average grain diameter is about 0.3 microns in the as-received condition. The microstructure of alloy 102 was very inhomogeneous and this is illustrated by the micrographs in fig. 3. There is a bimodal dispersoid size distribution in this alloy, and the distribution of dispersoids is nonuniform, leading to dispersoid poor regions.

Results from the TEM investigation indicate fairly large distributions in the dispersoid and grain sizes within a given specimen, making quantitative measurements laborious and difficult. The one set of quantitative results we would like to present concerns the effects of temperature and stress on grain coarsening. Figure 4 is a plot of grain size of alloy 102 samples which had been aged at 371°C, 482°C and creep tested (aged under stress) at 371°C with an applied stress of 60 MPa. Grain coarsening appears to occur in all three sets of experiments with 371°C aging exhibiting the least coarsening, 371°C with 60 MPa applied stress exhibiting slightly faster coarsening rates, and 482°C aging exhibiting the most coarsening. A word of caution however, although the mean values of grain size for all three conditions appear to increase with test time, the changes in mean grain size are small compared to the standard deviation of grain sizes within a given sample.

Discussion

The goal of the Navy sponsored HTA alloy development program was to develop a monolithic aluminum alloy for elevated temperature applications to replace heavier existing alloys, such as titanium alloys and steels, currently used to fabricate aircraft components subject to moderate elevated temperature operating conditions, resulting in weight, and possibly cost, savings. Starting with aluminum alloys FVS0812 (8009) and FVS1212, the objective was to develop an alloy which had higher strength and modulus than alloy FVS0812, and a higher room temperature ductility and fracture toughness than alloy FVS1212. Microstructurally, the alloy must be relatively stable at elevated temperatures 371°C.

The approach taken was to modify the alloy chemistry and dispersoid volume fraction; develop a series of experimental alloy compositions; and measure the room temperature mechanical properties of the experimental alloys. One of the experimental alloys developed under the HTA alloy development program which had the best combination of room temperature properties was selected to be the focus of the present work. This alloy, designated 102, had the

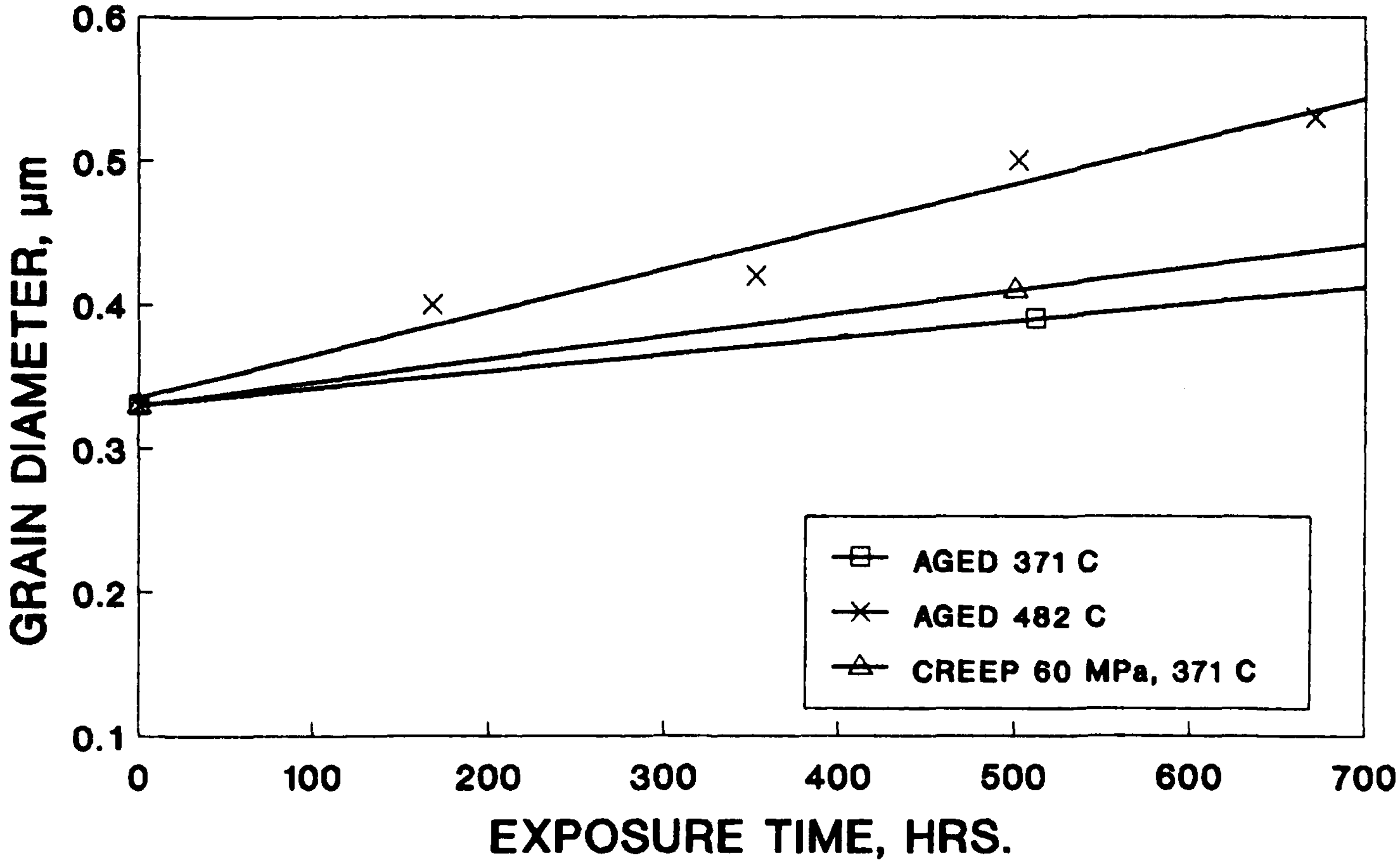


Figure 4 - Grain size of alloy 102 aged at 371°C, 482°C, and creep tested at 371°C with an applied stress of 60 MPa.

vanadium in the FVS-type alloys replaced with tungsten. The composition of alloy 102 is approximately 11.50% Fe, 3.75% W, and 2.25% Si by weight. This alloy has good room temperature strength 603 MPa while maintaining reasonable ductility (11.3% elongation to failure). The low diffusivity, limited solid state solubility, and high melting temperature of W lead one to anticipate slow dispersoid coarsening rates resulting in a relatively stable microstructure during elevated temperature exposure. The dispersoid volume fraction in alloy 102 is about 36%, and the Fe:W ratio is 9.2:1. Increasing the Fe:V ratio in the FVS-type alloys from 5:1 to 10:1 resulted in a factor of 3.5 decrease in dispersoid coarsening rates at 425°C [5]. The relatively high Fe:W ratio in alloy 102 would again lead one to expect slow dispersoid coarsening rates in this alloy.

The results from the creep testing performed on alloy 102 at 371°C were presented in fig. 1 and Tables I, II, and IV. The creep behavior of alloy 102 is fairly similar to that of alloy FVS1212 at 371°C. The alloy 102 creep curve exhibits a knee at an applied stress of about 80 MPa, corresponding to a minimum strain rate of approximately 1.5×10^{-9}/s. The stress exponent, n, increases from a value of about 1.5 for stresses less than 80 MPa, to a value of 12 at applied stresses greater than 80 MPa. This type of creep behavior is also seen in Al-Fe-Ce alloys. A knee in the Al-Fe-Ce creep strain rate vs. stress curve has been observed, and it moves to lower stresses and higher strain rates as the temperature is increased [7]. At a test temperature of 350°C, the knee occurs at about 45 MPa applied stress corresponding to a minimum strain rate of about 8×10^{-7}/s; the creep exponent at 350°C is equal to 1.7 at low stresses and 8.7 at high stresses [7].

Two data points from the alloy 102 creep testing results shown in fig. 1 do not lie close to the drawn curves. These points correspond to applied stresses of 69 and 77 MPa, very close to the knee in the curve, and the measured strain rates are higher than the corresponding points on the drawn curves. This positive deviation in minimum creep strain rates is probably a result of the inhomogeneous microstructure in this alloy and due to larger creep strains occuring in dispersoid poor regions of the sample.

The results from the aging and TEM studies on alloy 102 were presented in Tables V and VI and fig. 4. The room temperature hardness of alloy 102 is R_B 92 and does not change upon aging at 371°C for up to 1000 hrs. The hardness of alloy 102 did decrease upon aging at 482°C, however the change in hardness was relatively small for aging times up to 670 hrs. The hardness results suggest that the microstructure of alloy 102 is relatively stable with respect to long term elevated temperature exposure at

temperatures up to 482°C. TEM specimens were prepared from each of the aged samples and each of the creep tested samples. The goal was to measure the dispersoid and grain coarsening rates as a function of temperature and applied stress to determine if the microstructure of alloy 102 is indeed relatively stable, as the hardness results indicate. As mentioned previously, the inhomogeneous microstructure of alloy 102 does not permit quantitative measurements to be performed easily. The variations in grain and dispersoid sizes within a specimen were much larger than the expected changes in average sizes between specimens. Based on qualitative observations, the average grain and dispersoid sizes did increase with aging time; however, the increases were not large, even for the samples aged at 482°C for the longest times.

Conclusions

1. At 371°C, a change in creep deformation mechanisms occurs at approximately 80 MPa, defining the maximum practical operating stress for components made from this alloy.

2. There is a bimodal distribution of dispersoid and grain sizes in this heat of alloy 102. Also, the dispersoids are nonuniformly distributed through the material.

3. The room temperature hardness of alloy 102 did not change after aging at 371°C for greater than 1000 hrs indicating that the microstructure is stable at this alloy's intended use temperature.

4. Results from 482°C aging show a gradual decrease in hardness with aging time indicating that the microstructure is relatively stable even at this elevated temperature. TEM observations confirm that extensive coarsening does not occur in this alloy at temperatures up to 482°C.

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